Additional Questions Pertaining to CASC SOWs

SOM02.1 and ISM02.1 from Potential Offerors, with EPA Responses

October 24, 2013

SOM02.1

Exhibit C

Question 1: Table 3 (SV): Target analyte 4-chloroaniline, along with its DMC, 4-chloroaniline-d4, have been removed from Exhibit C.

Will a defect be generated by EXES if either the native or labeled analogs of this compound appear as extra peaks in a standard?

Answer 1: No defect will be generated if either the former target analyte 4-Chloroaniline or its deuterated analog is present in calibration standards used for the new contract. However, neither compound shall be calibrated or reported as a target analyte under the CASC.

Question 2: The CRQL of 2-Nitroaniline has been lowered to 5.0ug/L and 170ug/Kg. This compound is also a poor sensitivity one and was excluded in the low point calibration in SOM01.2.

Will this analyte be available at the new concentration in purchased ampulated standard mixes?

Answer 2: Target analyte 2-Nitroaniline has been shown to have sufficient sensitivity that it does not need to be classified as a poor performer. However, the ampulated standard mixes currently commercially available may contain this analyte at levels corresponding to the calibration requirements of SOM01.2.

Question 3: TCLP analytes are identified in Exhibit C with the same CRQLs for all fractions as normal water samples. It is not possible to overcome the matrix interferences found in most TCLP or SPLP leachate samples to report these CRQLs, whether or not there are any target analytes present. What can be done about this?

Answer 3: The SOW contains language that allows the contractor to perform appropriate dilutions for excessive matrix interference (10.2.4.1 of L/M VOA), or for repeatedly failing CCVs (10.4.4 of SV). If the semivolatile or pesticide extract exhibits matrix effects that preclude accurate quantitation of DMC or surrogate standards, the contractor would be expected to contact SMO to seek EPA permission to extract a smaller aliquot of leachate sample.

Exhibit D-SV

Question 4: DMC 4, 6-Dinitro-2-methylphenol-d2 is not included in 7.2.2.1.2, among the compounds requiring a higher calibration range (10, 20, 40, 80, 160ug/L) while its native

compound is. Therefore it is assumed that this DMC will be required to be calibrated at the lower range. Is this true?

Answer 4: Calibration standards for 4,6-Dinitro-2-methylphenol-d2 at either calibration range will be deemed acceptable, and will not result in the assessment of defects.

Question 5: For the initial calibration of semivolatiles, the required calibration levels of several of the analytes now labeled as poor performers in 7.2.2.1.2 will not be satisfied by making the customary five dilutions of a purchased ampulated stock standard.

Answer 5: Because of the changes made in developing SOM02.1, in Section 7.2.2.1.2, the statement above is true.

> **Question 5.1**: Given the current commercially available ampulated standard mixes for CLP, will the lab be allowed to use an alternate calibration procedure such as, for example, analyzing a series of six standards to cover all concentration levels? Will this procedure generate assigned defects in the electronic deliverable evaluation? Will the presence of extra analyte peaks in both the highest and lowest calibration levels generate any defects?
>
> **Answer 5.1**: A calibration procedure such as the example cited above would be acceptable. No defects would be generated during the CCS or EXES evaluations as a result of this calibration procedure.
>
> **Question 5.2**: Is there a plan to contact the standards vendors to encourage them to produce standard mixes that accommodate the new CLP calibration requirements?
>
> **Answer 5.2**: The vendors will be notified of the changes made to the CLP calibration levels.

Question 6: Continuing calibration requirements in D-SV.7.2.2.1.3 call for the CCV to be "at or near the midpoint concentration level of the calibration standards." If the lab were to use one of the currently available standard mixes, roughly half of the new "poor performers" would be at 40 ng/ml rather than at the midpoint for poor performers, which would be 20 ng/ml. Would this be acceptable?

Answer 6: The instruction at D.SV.7.2.2.1.3 states that the CCV standard should be ***at or near*** the mid-point concentration level of the calibration standards. With this instruction, the example cited above would be acceptable.

Question 7: SV section 11.1.2.5.7 requires the lab to report only one Tentatively Identified Compound match when the same CAS number is given for more than one chromatographic peak with a greater than 85% match. In work under the current contract, certain Regional customers have required the lab to report all matches over 85%. Would this generate a defect?

Answer 7: No, this would not generate a defect. However, if the mass spectral interpretation specialist feels that it is warranted to report the presence of multiple peaks represented in the search results by the same CAS number, this should be reported in the SDG narrative. Only one entry should appear on the Form 1B-OR per unique CAS number.

ISM02.1

Question 8: When reporting hardness, what FORM other than I is required?

Answer 8: Hardness is reported on Form 1-IN. On all other forms the Calcium and Magnesium data are used to evaluate if the various QC analyses met control limits etc.

Question 9: What M QUAL is used, I am assuming a 'P' because the result is derived from ICP AES analysis. Will hardness results only be derived from ICP AES analyses or would someone thing about using ICP MS results? If so would the M QUAL then be MS?

Answer 9: The "M" qualifier is no longer reported on Form 1-IN. Analytical Method is reported in the form header (Exhibit B/Section 3.3.9). Therefore, a separate Form 1-IN is to be submitted for each analytical method. Also, hardness is reported from the ICP-AES analytical method. A Modified Analysis would be needed to report hardness from ICP-MS.

Exhibit A:

> 5.4.1 If insufficient sample amount (less than the required amount) is received to perform the analyses, the Contractor shall contact SMO and proceed with the analysis of the sample at reduced volume. The Contractor shall document this action and the response from SMO in the SDG Narrative.

Question 10: Action states to use reduce volume. What volume should be used? Volume indicates that this should only apply to aqueous based samples. What about solid samples?

Answer 10: The volume used is somewhat dependent on the actual volume received. For example, if approximately one-half the volume necessary to perform the preparation is received, the lab is to use one-half the volume and adjust the reagents accordingly. This issue does tend to occur more often with aqueous samples, since preparations for a full soil analysis for all methods can be performed using less than 4 g of soil (plus some for percent solids). In cases where it is known in advance that sample amounts will be limited, the program has used Modified Analyses.

Question 11: What if sample preparation or analysis has begun by the time SMO responds and SMO responds not to prepare or analyze the samples in question? Is the laboratory compensated for the work they have already performed?

Answer 11: The compensation issue would be addressed by the Contracting Officer based upon the specific circumstances.

5.4.2 If the Contractor receives broken sample containers, with enough remaining sample to perform sample analysis, but potentially not enough volume to analyze any possible re-extractions/reanalyses, the Contractor shall note the issue in the SDG Narrative, proceed with analysis of the samples and notify SMO. If re-extraction/reanalyses are necessary, the Contractor shall contact SMO. The Contractor shall document the provided resolution in the SDG Narrative.

Question 12: I am assuming that SMO is only contacted if it turns out that there really is not enough samples to re-prepare or re-analyzed or if there is less than the nominal amount listed in Exhibit D. Is this correct?

Answer 12: The lab is to notify SMO in all cases, including when there is not a sufficient amount for any necessary re-preparations/re-analyses.

5.4.3 If the Contractor encounters other problems with samples or related documentation [e.g., mixed media, sample pH, sample documentation and paperwork such as Traffic Reports/Chain of Custody Records (TR/COC) not with shipment, sample and TR/COC do not correspond], the Contractor shall immediately contact SMO for resolution.

Question 13: I assume that this means that sample preparation and analysis should not start until SMO responds with a resolution. Is this correct?

Answer 13: Correct.

Question 14: How would a delay of a resolution impact required turnaround times?

Answer 14: The impact on turnaround times would be determined on a case-by-case basis depending on the timeliness of the resolution. SDGs with Preliminary Results and 7-day TAT requirements are expected to be the most severely impacted. In instances where a delay of resolution from the Region impacts contract required data delivery, the laboratory would need to request a waiver from their Regional Project Officer to adjust the data due date.

Question 15: Is the upper range (50^{o} C) of thermometer use to check the shipping container a little excessive.

Answer 15: The upper range is meant to cover possible situations, such as, where a lab during hot weather received samples that had been out on a sunny shipping dock all afternoon.

5.4.4.5 If a temperature indicator bottle is not present in the shipping container, and the temperature of the shipping container is less than or equal to 10°C, the Contractor shall note the issue, and the method used to determine the temperature, in the SDG Narrative and proceed with analysis of the samples. If the temperature exceeds 10°C and the samples are soil/sediment samples for any analytical method or aqueous/water samples for cyanide analysis, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the EPA for instructions on how to proceed. SMO will in turn notify the Contractor of the EPA's decision. The Contractor shall document the EPA's decision and the EPA Sample Numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

Question 16: I assume this would apply even if a temperature indicator bottle is present. Is this correct?

I assume that this means that sample preparation and analysis should not start until SMO responds with a resolution. Is this correct?

How would a delay of a resolution impact required turnaround times?

Answer 16: The requirements for samples exceeding 10°C apply in all cases, whether or not the cooler contained a temperature indicator bottle. See response provided for Section 5.4.3 above regarding the impact on contract turnaround times.

5.4.5.1 The pH for all aqueous/water samples received by the Contractor shall be measured, using a method capable of demonstrating that proper preservation was performed (e.g., pH test strips, electronic hand-held pen, pH meter), and recorded. The pH shall be determined using a small aliquot of the sample to prevent contamination. Under no circumstances shall a strip or any device be inserted into a sample bottle for the purpose of determining pH.

Question 17: How is the proper sample preservation recorded? Actual pH (e.g. 1.7, 12.3, 7.4, etc…) or in general terms (e.g. <2, >12, <12 and >2, etc…)

What is the proper course of action if the sample is not properly preserved?

Answer 17: The lab is to report the pH as the actual value measured. Instructions for samples that do not meet pH preservation requirements are in Exhibit D-Introduction/Section 5.0.

Question 18: What exactly is a TR/COC? The definition table at the beginning of Exhibit A lists COC and TR separately but no TR/COC.

I recall later in Exhibit A, instructions for filling in information on the TR or the COC and it did not make sense at the time. I will see if I can find that section.

Answer 18: The program usually uses a combined Traffic Report/Chain of Custody (TR/COC) form. The definition of this term is listed in the "Inorganic Abbreviation/Acronym List" below the Table of Contents.

5.5.3.2 If the Sampler designated two (or more) samples as QC for the same matrix, and the QC samples are not specifically labeled with the analysis they are to be used for (dissolved metals and total metals), then the Contractor is to contact SMO to report the issue. SMO shall then contact the EPA Region and notify the Contractor of the EPA Regional decision. If the Sampler did not designate QC samples, then the Contractor is to select a sample for QC and to contact SMO to report the issue.

Question 19: Last sentence: After selecting a sample for QC, what if sample preparation or analysis has begun and SMO responds back that the sample is not to be used?

Is the laboratory compensated for the work they have already performed?

Answer 19: The lab is not paid for matrix spike and duplicate sample analyses. Only if additional QC is required is the lab possibly eligible for additional payment. If the Region did not designate QC, any sample selected by the lab (excluding field blanks and PE samples) should be acceptable.

G.[7,8]	Determination of Method Detection Limits (MDL) And Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) Interelement Correction (IEC) Factors	1	MDL values in spreadsheet format specified in Appendix A of Exhibit H prior to analysis of field samples, annually thereafter, and after major instrument adjustments to SMO and QATS. MDL and IEC study data prior to analysis of field samples, annually thereafter, and after major instrument adjustments to QATS only. Submission of all deliverables within 7 days of determinations.	X		X

Question 20: Footnote seven, indicates that the results required in each CSF. Is this the information supplied on the FORMS 9 and 10? Or the actual study?

Answer 20: The results are to be reported on forms 9-IN, 10a-IN, and 10B-IN, as specified in Exhibit B/ Section 2.8.

Exhibit B:

> 3.3.7.2 These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if MA1111 is the lowest (considering both alpha and numeric characters) EPA Sample Number within the SDG, it would be entered in the first EPA Sample Number field. Samples would be listed below it, in ascending sequence - MA1111, MA1111D, MAB124, MAB125, MAC111, etc.

Question 21: The only FORM that I can think that would be list samples by EPA SAMPLE NUMBER is the SDG COVER PAGE.

All other FORMS only have one sample on them or need to be listed in chronological order. Is this correct?

Answer 21: Correct.

> 3.4.2.2.7 In the "Comments" field, note any significant changes that occur during sample preparation (e.g., emulsion formation), any sample-specific comments concerning the analyte results, and any raw instrument results that are less than minus the CRQL (-CRQL). These notes shall also be included the SDG Narrative.

Question 22: When documenting if the raw instrument result is less than the CRQL, the assumption is that the raw instrument concentration is compared to the base CRQL for water.

If a modified analysis (MA) is requested and the modification is a lower CRQL, does a new proportionate CRQL need to be calculated for comparison?

For example aluminum has a CRQL of 200 ug/L and 20 mg/Kg. If an MA was request with a CRQL or 100 ug/L or 10 mg/Kg, would the raw instrument concentration need to be compared to 100 ug/L or 200 ug/L?

Answer 22: Since raw instrument data is generally in aqueous units, the lab is to use the aqueous SOW CRQL when documenting results less than the CRQL. For Modified Analyses, the lab is to use the modified CRQL in all instances where the CRQL is to be compared to some other value. Therefore, for Modified Analyses, the lab is to use the modified aqueous CRQL when documenting results less than the CRQL.

3.4.2.2.7	In the "Comments" field, note any significant changes that occur during sample preparation (e.g., emulsion formation), any sample-specific comments concerning the analyte results, and any raw instrument results that are less than minus the CRQL (-CRQL). These notes shall also be included the SDG Narrative.
3.4.3.2.4.2	Under column "True", enter the expected concentration or true amount of each analyte in the CCV Solution.
3.4.3.2.4.3	Under column "Found", enter the concentration of each analyte measured in the CCV Solution.

3.4.2.2.7	In the "Comments" field, note any significant changes that occur during sample preparation (e.g., emulsion formation), any sample-specific comments concerning the analyte results, and any raw instrument results that are less than minus the CRQL (-CRQL). These notes shall also be included the SDG Narrative.

Question 23: I asked about this in a previous question submittal but do not think the question was fully addressed.

In the current ISM, we were told for the True Value to use the exact value listed (regardless of the number of sig figs) on the ICV or ICSA/B certificate received by the EPA.

True values for ICV and ICSA/B have been known to have more than two or three sig figs.

My understanding is that the true value should be populated using all sig figs supplied with the ICV or ICSA/B. Is this correct?

When populating the Found values, the 2 sig fig for <10 and 3 sig fig for greater >= 10 should be applied. Is this correct?

When calculating %R, the values on the FORM should be used. Is this correct?

Answer 23: The lab is to report True values as they are listed on the certificate of analysis and report Found values per the SOW requirements. The %Rs are to be calculated using these values and reported as specified in the Exhibit B, Section 3.4.3.2.4.4.

3.4.19.2.6	Under column "Weighting", enter the weighting factor for the calibration curve for each analyte calibrated in that analytical sequence. Report "Inverse Conc" for the inverse of the concentration; "Inverse Square" for the inverse square of concentration; or "None" if no weighting factor was applied.

I asked about this in a previous email but on review of Exhibit B, the weighting options are listed as:

9.4.4.2	The Contractor may use standard linear regression, weighted linear regression (e.g., 1/concentration or 1/concentration^2), or linear regression with zero force calibration models, as appropriate, for the above calculation. No other types of equations (e.g., quadratic) are to be used.

Question 24: The weighting option is read as "for example" and does not eliminate other weighting options.

I read in another question and answer session that a lab did not have these weight options with their Thermo ICP-MS software.

We also do not have those weighting options in our Thermo ICP-MS software.

Answer 24: The SOW requirements include the use of weighted linear regression, as provided in the example. ASB will review other possible options, as provided.